

MATERIALS SCIENCES DIVISION

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## Origin of Fatigue in Polycrystalline Silicon Determined

New Insight Could Prevent Premature Failure of Micromechanical Systems

The research team of C. L. Muhlstein and E. A. Stach and R. O. Ritchie has elucidated the mechanism by which thin films of polycrystalline silicon, identical to those used in most Microelectromechanical Systems (MEMS), can prematurely fail by fatigue when subjected to cyclic loading. The team used "on-chip" micro-testing machines, with dimensions of a few hundred micrometers, along with angstrom resolution and million-volt transmission electron microscopy at the National Center for Electron Microscopy to study these structures. They found that the origin of the fatigue failure is stress-assisted growth of a nanometer-thick surface layer of silicon nitride and moisture-induced cracking of that film. Limiting the formation of the oxide acts to minimize the susceptibility to fatigue failure.

Fatigue, the failure of a material at less than its initial ultimate strength after a number of cyclic loading cycles, is the most important and commonly encountered mode of failure in structural materials. After over a century of research, there are generally accepted mechanisms for the fatigue of ductile (e.g., metallic) and brittle (e.g., ceramic and ordered intermetallic) materials. However, although silicon is generally regarded as a prototypical brittle material, it lacks the three characteristics common to brittle materials that cause fatigue, namely, "dislocation motion," "grain bridging," and "microcracking." These considerations strongly imply that silicon should not fatigue in air at ambient temperatures, and, indeed, there is no evidence to date of bulk silicon being susceptible to fatigue failure under these conditions.

However, it was discovered in the early 1990's that thin silicon films do prematurely fail under cyclic fatigue loading in room-temperature air. The origin of these failures, however, has remained elusive until now, and although several mechanisms have been suggested, none have been supported by direct experimental evidence. Despite this, it has become increasingly important to understand this phenomenon, as over the past decade, MEMS, which incorporate silicon as a structural element, have evolved into established commercial products with applications ranging from optical switches that route Internet traffic to accelerometers in automobile airbags.

The study of fatigue on the scale of MEMS is challenging due to the small scale of the devices. In this study, MSD researchers used a highly sensitive testing system capable of measuring the fatigue of layers as thin as 2 micrometers (see figure). Samples were subjected to alternating tensile and compressive stresses at values below the initial failure strength of the material. By monitoring the natural frequency of the samples, the evolution of damage, in the form of nanometer-sized cracks growing from the notch, could be detected prior to the specimen's failing at between 10<sup>5</sup> and 10<sup>11</sup> cycles (higher loads produced failure within fewer cycles). Subsequent examination, using the Atomic Resolution (ARM) and the Kratos High-Voltage Electron Microscopes (operating at 0.8 – 1 MeV) at NCEM, of the partially and fully failed test samples, revealed that stress-assisted oxidation and moisture-induced crack growth within the nanoscale oxide layers that coat the silicon films were responsible for the observed fatigue behavior. The team further found that the coating of silicon with an alkene-based barrier layer of 1-octadecen, a hydrocarbon that is impermeable to water and oxygen, could vastly reduce susceptibility to fatigue failure. Such results are important from the perspective of increasing the durability and reliability of structural MEMS fabricated with silicon films.

R. O Ritchie (510 486-5798), Materials Sciences Division (510 486-4755), Berkeley Lab.

C. L. Muhlstein, E. A. Stach and R.O. Ritchie, "Mechanism of fatigue in micron-scale films of polycrystalline silicon for microelectromechanical systems," *Appl. Phys. Lett.* **80**(9) 1532-34 (2002) and C. L. Muhlstein, E. A. Stach and R.O. Ritchie, "A reaction-layer mechanism for the delayed failure of micron-scale polycrystalline silicon structural films subjected to high-cycle fatigue loading," *Acta Mater.* **50**, 3579-95 (2002).